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EXECUTIVE SUMMARY

LIF WASTE CHARACTERIZATION FOR HANDLING/DISPOSAL - 1985

A. Golomb

Preparatory to a trial of direct limestone injection into the furnace (LIF) for acid gas control, to be held at Lakeview TGS in November/December, 1985, laboratory tests have been carried out to anticipate the physical and chemical characteristics of the solid waste produced, to facilitate safe handling and disposal. A sample of Consol LIF waste, obtained from the USA, was found to have a CaO content of 333 g/kg; in calorimetric tests it reacted exothermically with water ($\Delta H \approx -68.9$ cal/g). Leachate derived from the 'as received' waste had pH 12.4. In bulk slaking tests, internal temperatures up to 160°C were noted, accompanied by ~15% linear expansion. Particle size analysis showed the slaked material to be finer than the original waste. The need for care in handling and disposal of LIF waste is therefore evident. A block experiment has been carried out to predict leaching characteristics of the landfilled waste prepared using various levels of added water, added fly ash, and curing times at summer and winter temperatures. Standard MOE leachate extraction tests and detailed physical tests indicate that the fixated waste is essentially non-hazardous and suitable for use as landfill or backfill. Based on the results of experimentation with Consol LIF waste, recommendations are made for future work.

job	file	date	report no.
740631-232-901	837.2412	January 10, 1986	85-340-K

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LIF WASTE CHARACTERIZATION FOR HANDLING/DISPOSAL - 1985

A. Golomb

To facilitate safe handling and disposal of solid waste resulting from a scheduled trial of acid gas control by limestone injection into the furnace (LIF) at Lakeview TGS, preliminary tests were carried out on a sample of Consol LIF waste obtained from the USA. The Consol waste had a CaO content of ~333 g/kg, and reacted exothermically with water ($\Delta H \approx -68.9$ cal/g). Leachate derived from the 'as received' Consol waste had pH 12.4. In bulk slaking tests, internal temperatures of 160°C were observed; this was accompanied by ~15% linear expansion. Particle size reduction on slaking was also noted. The need for care in handling and disposal of LIF waste is therefore evident. Standard MOE leachate extraction tests have been carried out on the 'as received' and fixated Consol LIF waste, and physical/engineering properties measured to determine suitability for disposal as landfill. The results are promising. Recommendations are made for future work.

INTRODUCTION

Ontario Hydro plans to determine the feasibility of acid gas emission control by direct injection of limestone into the furnace (LIF). Tests are scheduled to take place at Lakeview TGS in November/December, 1985. It has been suggested that handling and disposal of the resultant limestone-modified fly ash (LIF waste) could pose problems due to the expected high CaO content. Such problems might include:

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(i) exothermic reaction of the waste (containing up to 40% w/w of calcined limestone, ie CaO) when wetted,

(ii) potential for pozzolanic reaction of mixtures of fly ash and CaO with water,

(iii) leachate problems that might occur in relation to landfill disposal

and

(iv) dusting problems arising from handling/disposal of the waste when dry.

A brief, exploratory LIF test was carried out at Lakeview TGS, on January 29-30, 1985, resulting in production of about 300 Mg of waste, which was hauled by tanker truck for temporary dry storage in a silo at Hearn TGS. (After investigation by a specially-appointed task force, this material, which had been diluted with regular fly ash, was later safely and effectively disposed of (a) via the ash sluicing system at Nanticoke TGS, and (b) by landfill at the Brittania Road municipal dump site, in the County of Peel.)

WORK PROGRAM

To generate useful data for design and development of safe, effective methods for handling and disposal of LIF waste to be produced at Lakeview TGS in November/December, 1985, and at other coal-fired stations in the future, the research program outlined below was planned and executed. The LIF material selected for initial testing purposes was Consol limestone injection waste, obtained from recent tests using eastern US bituminous coal (sulphur content ~2.5% w/w) at the DuPont Inc nylon production plant at Martinsville, Virginia, USA. Consol waste was considered to be the best available substitute for the November/December, 1985, Lakeview LIF waste, since it was known to have a high CaO content resulting from calcination of limestone and a modest degree of sulphation.

1. Initial Waste Characterization

   - chemical analysis, exothermicity measurements, particle size determination, and MOE leaching tests

2. Waste Fixation/Screening

   - preliminary setting tests with combinations of Consol waste, Lakeview TGS fly ash, and water - followed by waste fixation experiments and screening to identify the four best candidates for further, more detailed testing

3. MOE Leachate Extraction of Selected Fixated Waste

(a) using deionized water and
(b) with pH adjustment in accordance with the 1984 Leachate Extraction Procedure/1,2,3/.

4. Engineering/Physical Tests of Selected Fixated Waste

- further engineering tests (eg compressibility, permeability etc) of selected fixated waste samples to check suitability for landfill use.

5. Slaking/Pelletizing Equipment Evaluation

- samples of Consol waste to be sent to commercial equipment suppliers for evaluation.

PROGRESS TO OCTOBER, 1985

Chemical Analysis

The major chemistry of Consol LIF waste has been determined. Analytical results are given in Table I. For comparison, available chemical analyses of LIF waste sampled during the January, 1985, exploratory test at Lakeview TGS are included. CaO content was determined for 10 individual tanker-loads of LIF waste leaving Lakeview TGS for storage in a silo at Hearn TGS. The highest CaO tanker load was identified. A composite sample was also made up from the 10 tanker-loads. LIF waste samples were taken during the test from a dedicated electrostatic precipitator hopper in the morning and afternoon of both January 29 and 30, 1985. From Table I, it is apparent that the Consol waste and the hopper waste samples (which had the highest CaO content) should be more representative of the LIF waste expected to be generated in the fall, 1985, test than any of the tanker waste samples. There is evidence that CaO in the tanker-truck LIF waste was diluted to varying degrees by regular fly ash formed during the test run (i) at times when limestone injection rates were low or nil and (ii) by fly ash originating in the other boiler unit connected to the common silo from which the tankers were loaded.

The highest CaO content of the hopper samples (331 g/kg) occurred on the afternoon of January 29, 1985, when limestone injection rates reached their peak.

The $CaSO_4$ content of the Consol waste was about three times higher than that of any of the Lakeview January test samples: this may reflect poor sulphation efficiency in the Lakeview January test, which could in turn be due, at least partly, to dead-burning.

TABLE I

CHEMICAL ANALYSIS OF CONSOL LIF WASTE AND LAKEVIEW TGS (JANUARY, 1985) LIF WASTE

Species	Consol LIF	LVTGS 10-Tanker Composite	LVTGS High-CaO Tanker	LVTGS Hopper LIF 840129 AM	LVTGS Hopper LIF 840129 PM	LVTGS Hopper LIF 840130 AM	LVTGS Hopper LIF 840130 PM
CaO	333 (270)	75	160	240	331	278	268
$Ca(OH)_2$	- (<30)	(<30)	(<30)	(<30)	(<30)	(<30)	(<30)
$CaSO_4$	182 (190)	35	51	61	55	55	52
$CaCO_3$	80 (50)	8	12	(<30)	(<30)	(<30)	(<30)
Unburnt Carbon	129	86	92	-	-	-	-

All concentrations given in g/kg. Results of analysis by XRD are given in parentheses.

If we assume that the LIF waste to be produced in the fall,1985, Lakeview test will resemble the Consol material, safety precautions will have to be taken with respect to the expected high CaO content. Dusting must be avoided during handling operations, and the waste carefully and efficiently slaked prior to disposal, eg as landfill.

Exothermicity Tests

The heat of reaction of Consol waste with water was measured using a styrofoam-jacketed nickel calorimeter and stirrer of total water equivalent 10.9 g. The stirrer, which had a rotational speed of 60 rpm, was used for the first 1 min of each run, and then switched off to minimize frictional heat. Forty-gram quantities of Consol waste were allowed to react with various amounts of water, and the exothermic reaction temperatures plotted versus time. For this purpose, a copper-constantan thermocouple was used for temperature measurement. A Hewlett-Packard HP-85 microcomputer and a HP-3421A data acquisition/control unit were used for data collection/reduction.

Results are given in Figure 1, which shows that the maximum temperatures observed are inversely related to the amounts of water added. (This is as expected, since at higher liquid/solid ratios the heat of reaction is dissipated into a larger 'heat sink'.) The highest temperature reached in these tests (~100°C) was for 10 g water added to 40 g Consol LIF waste; water vapour condensed on the cooler parts of the apparatus under the conditions. To measure the heat of reaction, temperature/time curves corresponding to larger amounts of added water (ie 35 g, 45 g, and 60 g water added to 40 g Consol waste) were selected for use. Applying a cooling temperature correction/4/ for cooling by projecting the near-linear cooling section of each plot back to time zero, values were calculated for the heat of reaction of Consol waste with water; results are given in Table II.

The average calculated heat of reaction of Consol waste with water ($\Delta H \approx -68.9$ cal/g) is equivalent to about 30% of the heat of slaking of quicklime ($\Delta H \approx -230$ cal/g). This is in good agreement with the observed 33.3% of CaO (27.0% by XRD) found in Consol waste. The heat of slaking of CaO is known/5/ to be liberated much more rapidly than the heat of hydration of anhydrite ($\Delta H \approx -30$ cal/g) - the most probable form of $CaSO_4$ in Consol waste. Since strong, concrete-like setting was <u>not</u> observed in the cured material (it was friable) it is considered improbable that the heat of bonding between lime and fly ash could have contributed much heat to that observed. In summary, the observed heat of reaction between Consol LIF waste and water is attributed predominantly to the slaking of CaO.

LIMESTONE INJECTION FURNACE

CALORIMETRY: TEMP (C) VS TIME (SEC)

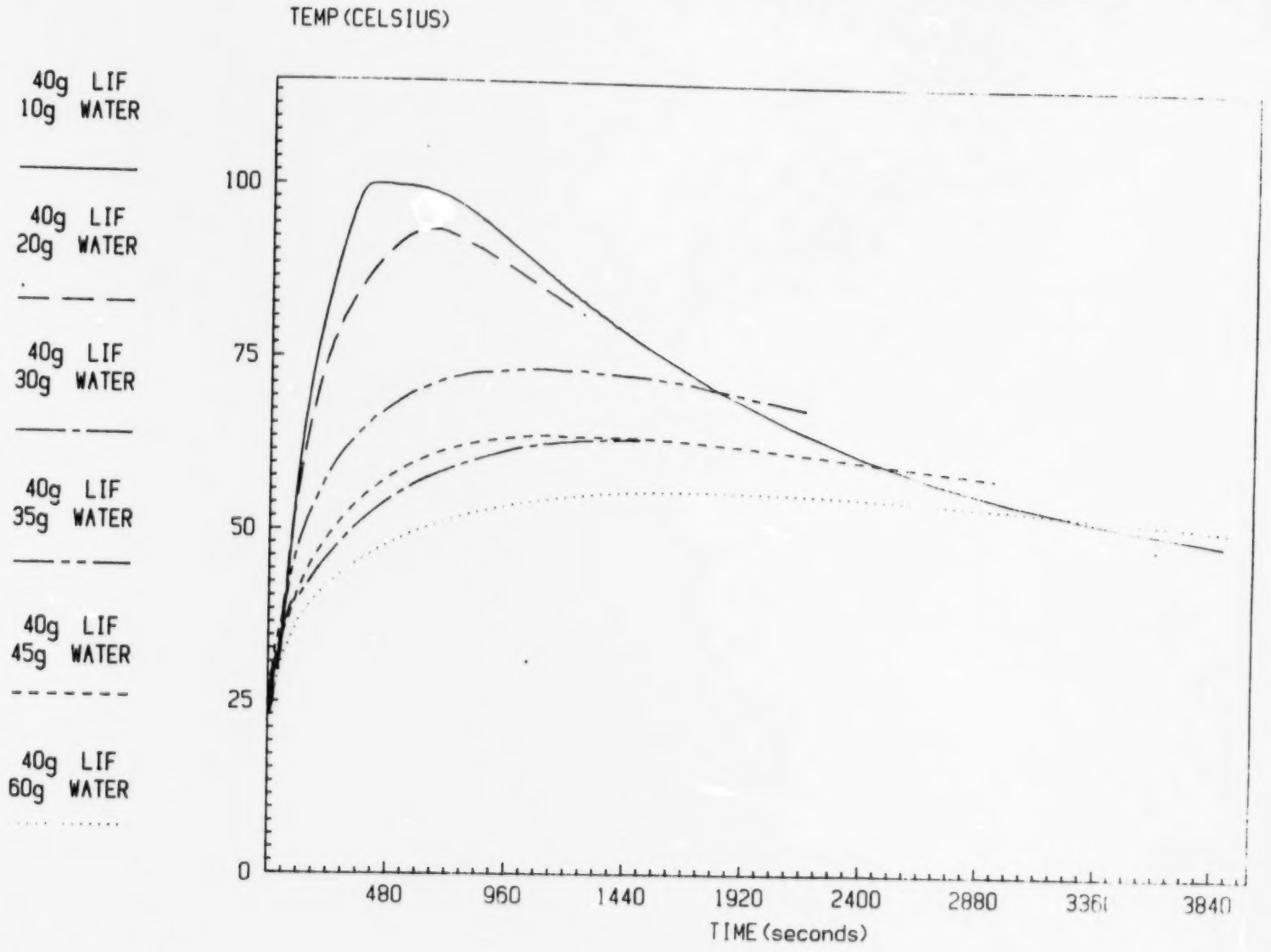


FIGURE 1

TABLE II

CALCULATED HEAT OF REACTION OF CONSOL LIF WASTE WITH WATER

Wt of Consol (g)	Wt of H_2O (g)	Temp Projected Back to Time 0 (°C)	Ambient Temp (°C)	Water Equivalent of Calorimeter (g)	ΔH (cal/g)	
40	35	84.1	23.5	10.9	-71.4	
40	45	70.2	23.5	10.9	-66.6	Av = -68.9
40	60	61.5	23.5	10.9	-68.6	

Exothermicity tests were also carried out on samples of LIF waste generated during the Lakeview January trial. Results are given in Table III.

Although CaO analysis of Consol LIF and hopper LIF (January 29, PM) wastes were about the same (~33% w/w) the hopper material generated far less heat on slaking. The hopper material may well have been dead-burned, which would account for its relative lack of reactivity. During the January test, limestone was injected at the top row of burners, where temperatures were well in excess of 1 050°C (the commonly-accepted threshold temperature for dead-burning).

Bulk Exothermicity Test

For safety reasons, it was of interest to measure the maximum temperatures attainable within a mass of LIF waste when exposed to small amounts of water during bulk handling or storage, ie. where mechanical mixing action is practically negligible.

About 2 L of Consol LIF waste was lightly packed into a cylindrical container of rigid styrofoam (internal dia 14.0 cm, height 15.5 cm), fitted with a rigid styrofoam lid. The underside of the lid was equipped with a water distribution coil, made of perforated silicone tubing. Deionized water was pumped from a reservoir via a calibrated Cole-Parmer peristaltic pump at ~3.7 mL/min over the top of the bulk Consol waste, and allowed to percolate down through the material. This application rate corresponds to a rainfall of ~1.63 cm/h which, based on meteorological data, is possible for southern Ontario. Three thermocouple probes were located axially at 10 cm, 7 cm and 4 cm above the base of the container. As the water front passed downward, the rise in temperature at each probe was measured and recorded (Figure 2). The maximum temperature observed within the bulk material was ~160°C. This is higher than the ~100°C maximum observed in the most strongly exothermic calorimetry test. The high bulk slaking temperature is attributed to poor thermal conductivity of the LIF waste, which did not effectively dissipate the heat. The final, slaked bulk material was found to have expanded by ~15%, ie. from 14.0 to 16.2 cm dia at the middle. In practice this expansion could damage or rupture containing vessels, eg tankers, silos etc, unless steps are taken to avoid uncontrolled water addition during handling.

If the waste were landfilled dry (ie unslaked), moreover, expansion inevitably resulting from infiltration of water would probably result in heaving and fissuring, with attendant loss of structural integrity, as well as creating steam and dusting problems in the vicinity.

TABLE III

EXOTHERMIC REACTION TEMPERATURE OF LAKEVIEW (JANUARY, 1985) LIF WASTES WITH WATER

Waste	CaO Content (% w/w)	Maximum Temperature (°C) Observed	
		40 g Solid + 10 g Water	40 g Solid + 30 g Water
Hopper LIF 850129 AM	24	38.4	29.4
Hopper LIF 850129 PM	33.1	51.2*†	51.5*†
Hopper LIF 850130 AM	27.8	34.3	28.3
Hopper LIF 850130 PM	26.8	35.6	29.8
10-tanker composite	7.5	29.0	25.2
High-CaO tanker	16.0	28.4	26.7
Consol LIF (for comparison)	33.3	~100†	71.2†

* Anomalous equality of maximum observed temperatures (both were re-checked).
† Maximum temperatures lower for Hopper material than for Consol waste

CONSOLIDATED WASTE
BULK EXOTHERMIC STUDY

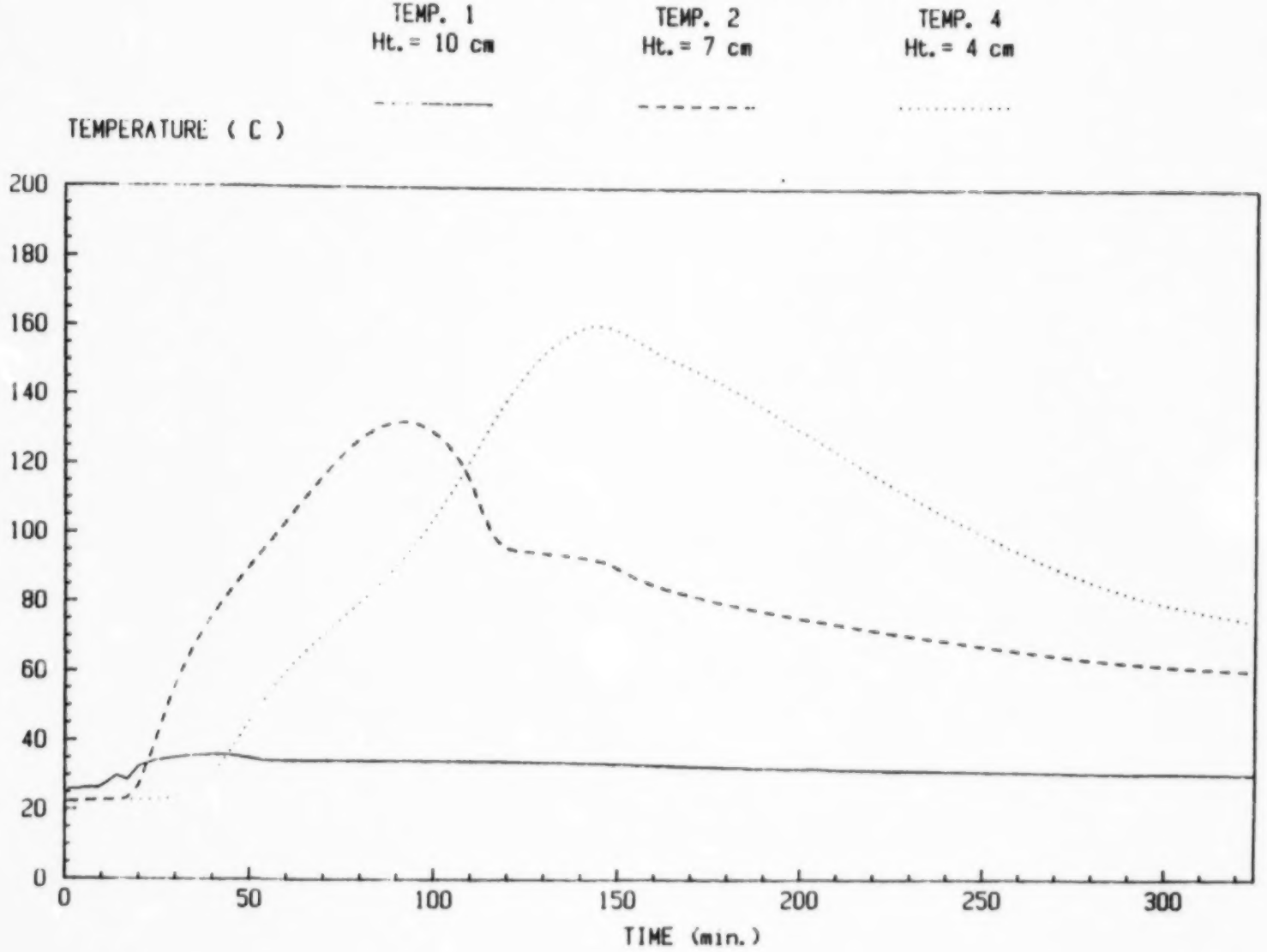


FIGURE 2

Particle Size Determination

Particle size distribution measurements were made using a HIAC Particle Size Analyzer (Model PC-320). Determinations were made on Consol LIF waste 'as received' and on a sample of the dry, slaked material remaining after reacting 40 g Consol waste with 10 g of water (Figure 1, highest exotherm). Results are given in Table IV.

TABLE IV

HIAC PARTICLE SIZE ANALYSIS OF CONSOL WASTE 'AS RECEIVED' AND AFTER SLAKING

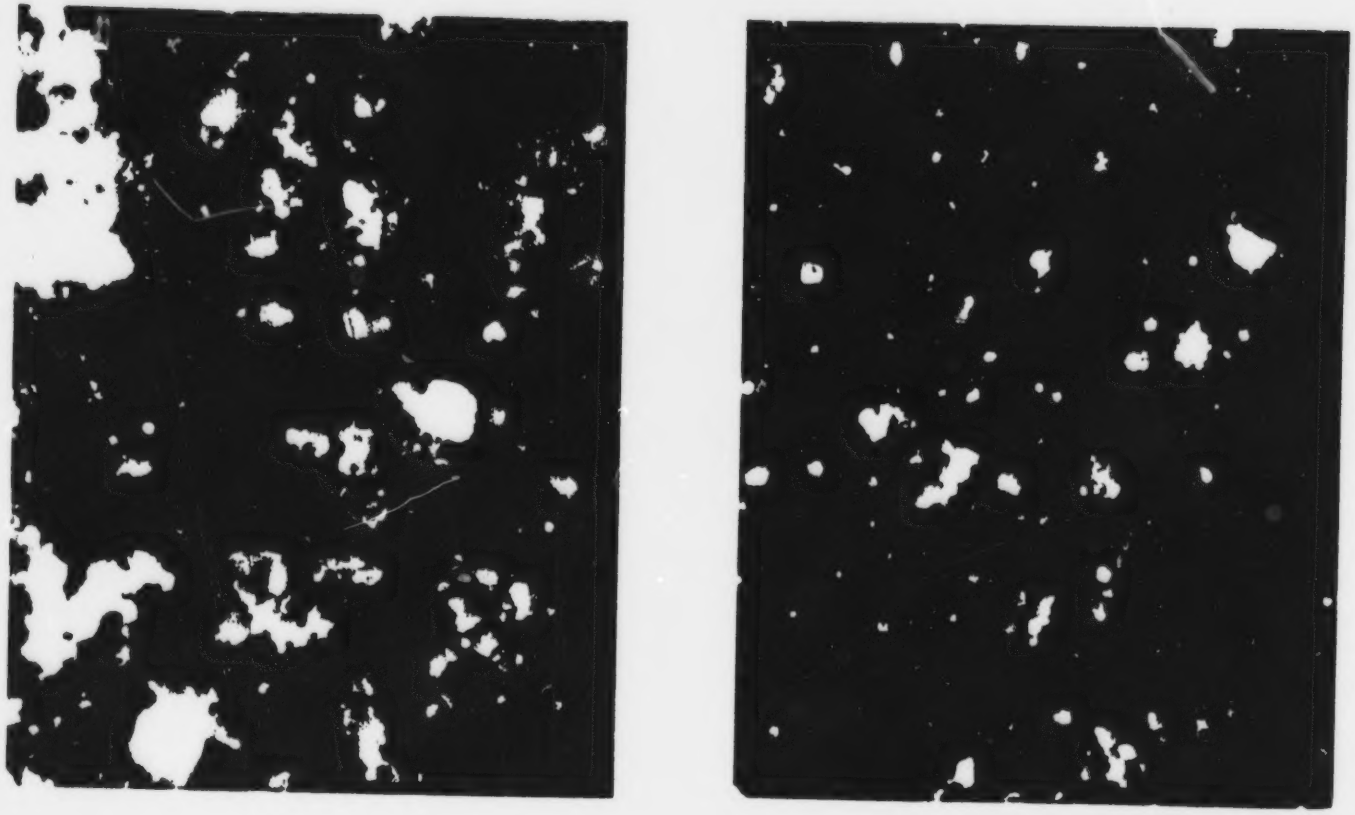
Size Range (μm)	No. of Particles/mg Sample*	
	'As Received'	Slaked
5 - 10	33 550	46 800
25 - 50	2 000	7 150
50 - 100	362	770
>100	32	52

*Note: Increase of number of particles per mg sample in all size ranges is attributed to exothermic (slaking) chemical reaction, in which heat and steam evolved causing breakdown of all reactive CaO particles.

It is seen from Table II that the material became finer when slaked. This observation is supported by optical microscopy (Figure 3) at 90X magnification. The heat of reaction was sufficient, moreover, to evaporate the remaining water and leave a dry residual product. For handling/disposal of slaked LIF waste, it is essential that appropriate steps be taken to avoid dusting problems associated with the finely-divided material.

MOE Leaching Tests

A set of leaching tests was carried out to determine the leaching characteristics of the 'as received' Consol waste. The test procedure used is essentially that proposed by the MOE in October, 1984/1,2/. To gain maximum information, leaching tests were conducted under aerobic as well as anaerobic conditions, with and without addition of acetic acid for pH adjustment.



(a) (b)

FIGURE 3

Consol Waste: (a) 'as received', (b) after slaking

(90X magnification)

Results are reported in Table V. The extract obtained using deionized water had pH 12.4 at 22°C. Chemical species analyzed in the leachates are: As, Ba, B, Cd, Cr, W.A.D.[1] CN^-, F^-, Pb, Hg, NO_3^- (as N), NO_2^- (as N), Ra-226, U, Se, Ag, Cl^-, Cu, Fe, Mn, N (organic), phenols, SO_4^{2-}, TDS, TOC, PCBs, and Zn. Of these, barium and boron just exceeded the backfill criteria. TDS levels exceeded the backfill requirement (~4500 versus 2500 mg/L), attributable mainly to $Ca(OH)_2$ and $CaSO_4$ - components of calcium hardness in water. From Table V it is also evident that the 'as received' Consol LIF waste is non-hazardous based on MOE leaching characteristics.

Waste Fixation - Preliminary Tests

To gain some idea of the physical handling characteristics of LIF waste and of LIF waste/fly ash after mixing with water, a series of preliminary tests was carried out. It was hoped that the results would provide a key to selection of conditions for rendering the waste suitable for landfill disposal.

Samples of Consol LIF waste after exothermicity testing (slaking) were removed from the calorimeter and air-dried in the open laboratory at 23-24°C for 24 h (Figure 4). As previously noted, the basis of these tests was 40 g of dry Consol material; amounts of water added were 10 g, 20 g, 30 g, 35 g, 40 g, 50 g and 60 g, respectively. It was found that water additions of 10 g, 20 g and 30 g gave products ranging from dry, to moist, to moist/lumpy. Water addition of 35 g gave a smooth paste that set lightly over 48 h to a plaster-like solid. Higher levels of added water resulted in phase-separation; solid material settled out under an aqueous supernatant layer. When the excess water evaporated off at room temperature (~48 h) the solid material set lightly, with a weak crusty layer on top, resembling 'dried mud'.

Samples of Consol LIF waste were mixed with various proportions of added Lakeview fly ash (total weight of dry solids = 40 g) covering the entire range from 100% fly ash to 100% Consol LIF waste with seven intermediate blends. Water was thoroughly blended with each mix to a wet plaster-like consistency, which (except for 100% fly ash) took on a moderate set in the open laboratory after 24 h, during which time some drying-out took place (Figure 5). The maximum temperatures observed in these open-laboratory tests were, as expected, considerably lower due to heat loss than those noted earlier in the calorimetry experiments. The various conditions and corresponding open-laboratory temperatures reached are summarized in Table VI.

[1] W.A.D. CN^- denotes weak acid dissociable cyanide.

TABLE V

MOE LEACHATE EXTRACTION: ANALYTICAL RESULTS[a]

FOR CONSOL LIF WASTE SOLID 'AS RECEIVED'

Parameter	Monofill		Co-Disposal		Backfill Use: Maximum Acceptable Extract Concentration (Monofill Test)/1,2,3/	Hazardous Waste Categorization: Maximum Extract Concentration For Exemption (Co-Disposal Test)/1,2/	Comments
	Aerobic	Anaerobic	Aerobic	Anaerobic			
pH	12.4	12.4	12.5	12.5	-	-	No significant pH change
As	<0.001	<0.001	<0.001	<0.001	0.05	5	
Ba	1.1	2.0	1.1	3.0	1.0	100	Ba slightly exceeds backfill criteria
B	5.4	5.5	5.6	5.7	5.0	500	B slightly exceeds backfill criteria
Cd	0.0001	<0.0001	<0.0001	<0.0001	0.005	0.5	
Cr (total)†	<0.1	<0.1	<0.1	0.1	0.05	5	Cr detection limit being improved
'WAD' (CN)	<0.04	<0.04	0.05	0.04	0.2	20	
F⁻	0.7	0.4	0.98	1.0	2.4	240	
Pb	<0.002	<0.002	<0.002	<0.002	0.05	5	
Hg	<0.0002	<0.0002	<0.0002	<0.0002	0.001	0.1	
NO_3 (as N)	0.22	0.22	0.17	0.19	10.0	1000	
NO_2 (as N)	0.05	0.05	0.03	0.05	1.0	100	
Total [NO_3^- + NO_2^-] (as N)	0.27	0.27	0.20	0.24	10.0	1000	
U †	<0.02	<0.01	0.03	0.02	0.02	2	U detection limit being improved
PCBs	<0.0002	<0.0002	0.0006	0.0006	0.003	0.3	
Ra-226	<0.5	<0.5	0.6	0.6	3.0 pCi/L	300 pCi/L	
Se	0.012	0.014	0.013	0.014	0.01	1	
Ag	<0.0002	<0.0002	<0.0002	<0.0002	0.05	5	
Cl^-	136	142	136	138	1250	-	
Ca	<0.05	<0.05	0.07	0.09	5.0	-	
Fe	<0.5	<0.5	<0.5	<0.5	1.5	-	
Mn	<0.05	<0.05	<0.05	0.08	0.25	-	
Organic N	<0.06	<0.06	<0.06	<0.06	0.75	-	
Phenols	<0.001	<0.001	<0.001	<0.001	0.01	-	
$SO_4^=$	1880	1210	1290	1675	2500	-	
TDS	4800	4160	N/A	N/A	2500	-	Observed TDS levels exceed 2,500 mg/L
TOC	<1	1	N/A	N/A	25	-	
Zn	0.02	<0.04	0.02	0.04	25	-	

a All concentrations given in mg/L except Ra-226, given in pCi/L.

† Analytical interference due to high background Ca concentrations.

N/A Denotes not applicable due to acetic acid addition in co-disposal test.

FIGURE 4

Consol Waste/H_2O - Preliminary Fixation Samples

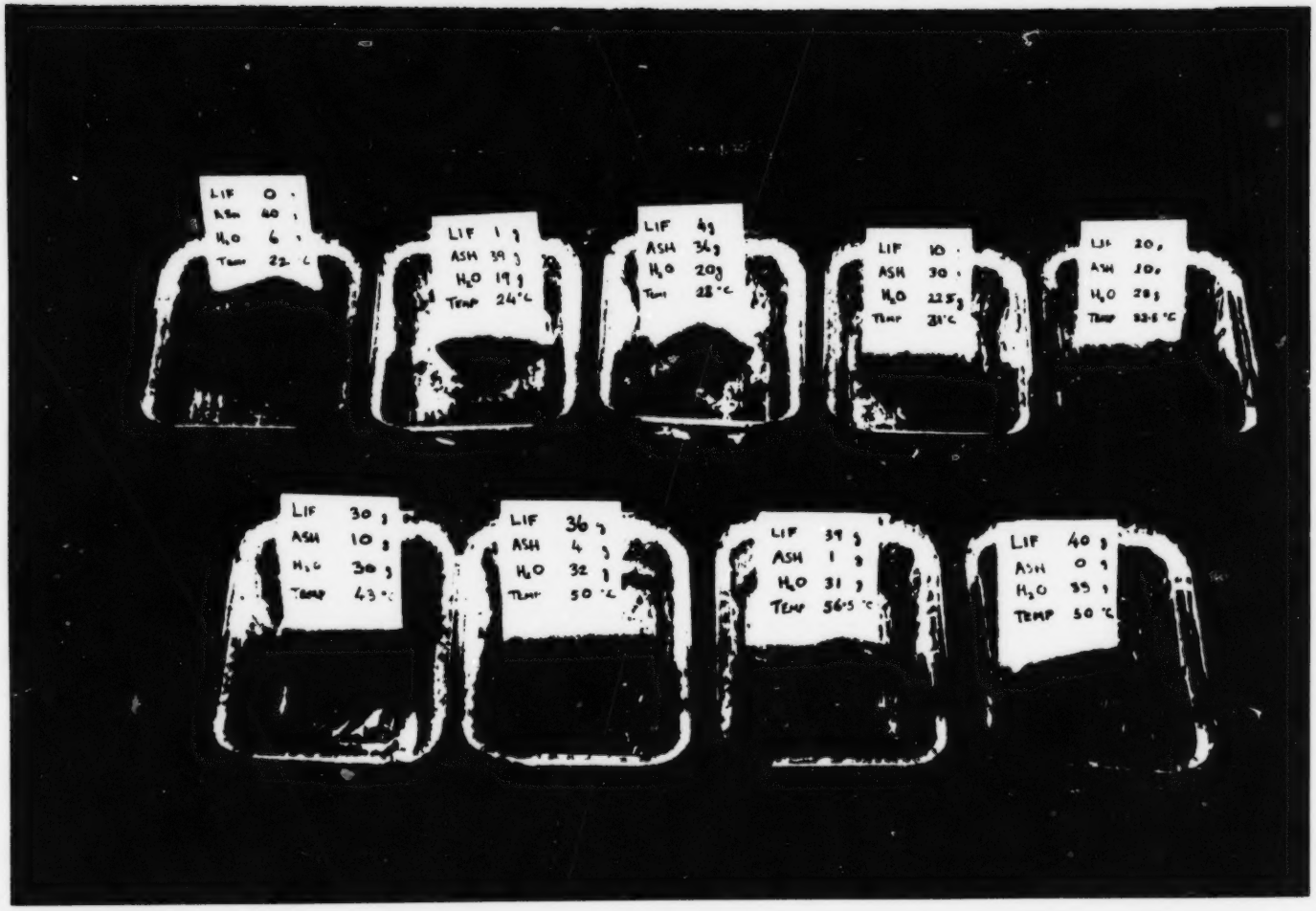


FIGURE 5

Consol Waste/Fly Ash/H_2O - Preliminary Fixation Samples

TABLE VI

BLENDS OF CONSOL LIF WASTE AND ADDED LAKEVIEW TGS FLY ASH.
OPEN-LABORATORY MAXIMUM TEMPERATURES ATTAINED

Consol LIF Waste (g)	0	1	4	10	20	30	36	39	40
Added Lakeview Fly Ash (g)	40	39	36	30	20	10	4	1	0
Water Added to 'Smooth Paste' (g)	6	19	20	22.5	28	30	32	31	33
Maximum Temperature Attained (°C)	22	24	28	31	32.5	43	50	56.5	50

To obtain a plaster-like set from Consol LIF/fly ash mixtures containing more than 50% w/w of Consol material (dry solids basis) it was found necessary to add 30 - 33 g water per 40 g of dry solids.

Samples of Consol waste and Lakeview fly ash were mixed to simulate the waste material produced at Ca/S ratios of 2.5 and 1.25 in Unit 4 operating under LIF conditions, with Unit 3 generating fly ash normally, the two units feeding into a common silo. This scenario was planned to anticipate conditions for the January, 1985, Lakeview test. For each Ca/S ratio, water was added corresponding to 5%, 10% w/w and higher levels of water on a dry solids basis. Addition of increasing amounts of water to 40 g dry solids resulted in formation of dry to powdery-moist products for up to 29 g of added water, at which level setting to a plaster-like product took place (Figure 6). The various conditions and corresponding open-laboratory temperatures reached are summarized in Table VII.

Waste Fixation - Block Screening Experiment

A block experiment was carried out to prepare fixated samples of varying proportions of Consol LIF waste, added Lakeview fly ash, and moisture content; and at different curing temperatures and curing times.

The samples prepared as detailed in Table VIII were subjected to accelerated screening/leaching tests under monofill aerobic conditions (no pH adjustment; air in headspace). Preliminary

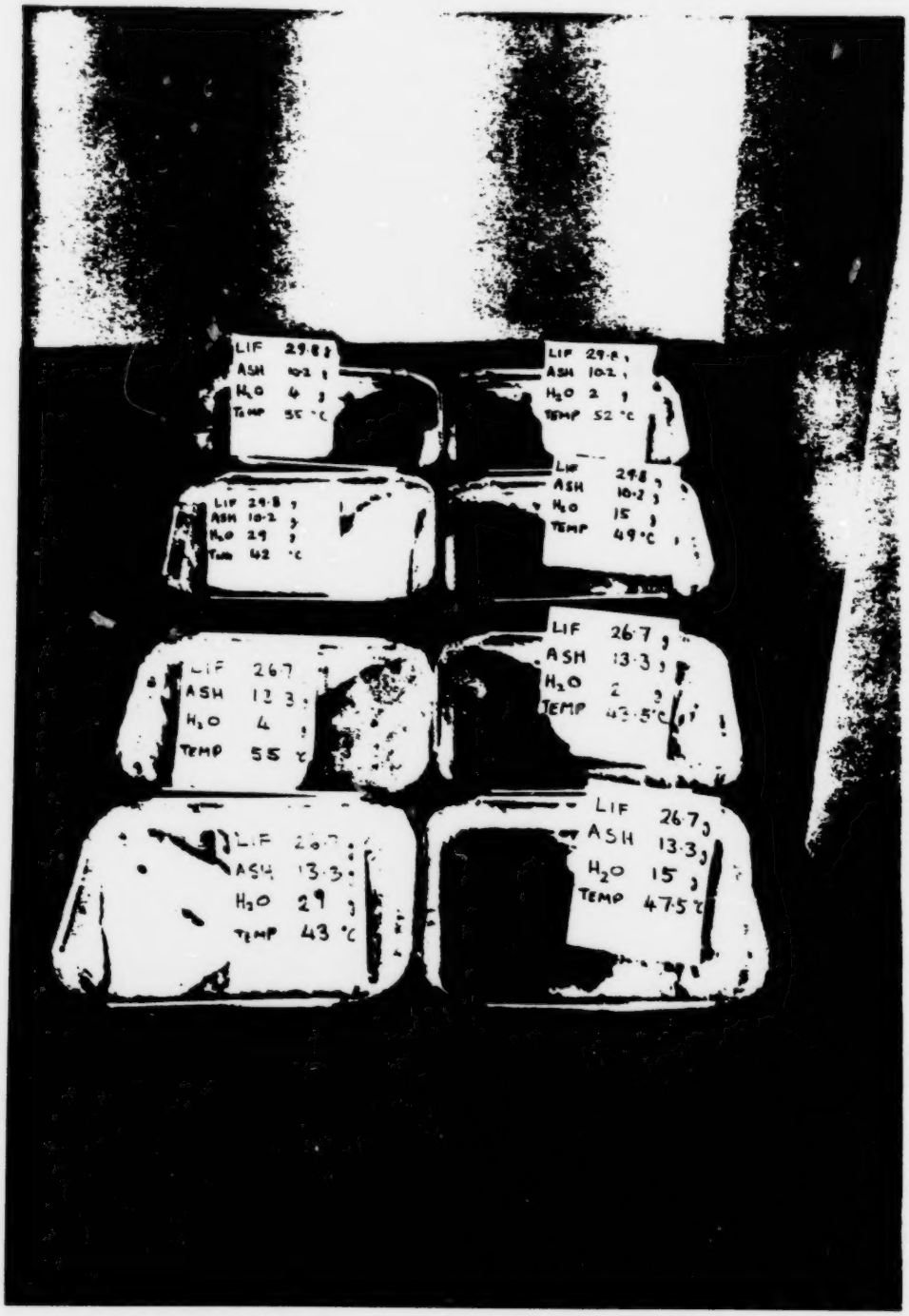


FIGURE 6

Ca:S Ratios 2.5 and 1.25 - Consol Waste/Ash/H_2O - Preliminary Fixation Samples

TABLE VII

BLENDS OF CONSOL LIF WASTE AND LAKEVIEW TGS FLY ASH.
SIMULATION OF Ca/S RATIOS 2.5 AND 1.25 IN UNIT 4 WITH
UNIT 3 OPERATING NORMALLY. MAXIMUM TEMPERATURES ATTAINED

Ca/S = 2.5	Consol LIF Waste (g)	29.8	29.8	29.8	29.8
	Added Lakeview Fly Ash (g)	10.2	10.2	10.2	10.2
	Water Added (g)*	2 (5%)	4 (10%)	15 (37.5%)	29 (72.5%)
Ca/S = 1.25	Consol LIF Waste (g)	26.7	26.7	26.7	26.7
	Added Lakeview Fly Ash (g)	13.3	13.3	13.3	13.3
	Water Added (g)*	2 (5%)	4 (10%)	15 (37.5%)	29 (72.5%)

* Figures in parentheses denote percentage water added (dry solids basis).

TABLE VIII

CONSOL LIF/FLY ASH/WATER MIX PROPORTIONS, CURING TIMES & TEMPERATURES, AND SCREENING TEST RESULTS

Sample Reference Number	LIF Waste (Consol) (g)	Fly Ash (Lakeview TGS) (g)	Water (Toronto Tap) (g)	Curing Temp. (°C)	Curing Time (days)	Screening Extraction Test Concentrations (mg/L)		
						B	Se	As
S-1A	40	0	30	22	7	0.079	0.018	<0.001
S-1B††	40	0	30	22	28	0.110	0.007	<0.001
W-1A	40	0	30	-5	7	2.97	0.008	<0.001
W-1B	40	0	30	-5	28	1.71	0.015	<0.001
S-2A†	40	0	32.5	22	7	0.110	0.006	<0.001
S-2B	40	0	32.5	22	28	0.13	0.006	<0.001
W-2A	40	0	32.5	-5	7	2.81	0.015	<0.001
W-2B	40	0	32.5	-5	28	1.46	0.009	<0.001
S-3A	40	0	35	22	7	0.092	0.008	<0.001
S-3B	40	0	35	22	28	0.067	0.008	<0.001
W-3A	40	0	35	-5	7	2.95	0.011	<0.001
W-3B	40	0	35	-5	28	1.76	0.010	0.001
S-4A	29.8	10.2	30	22	7	0.094	0.009	<0.001
S-4B*	29.8	10.2	30	22	28	<0.05	0.003	<0.001
W-4A	29.8	10.2	30	-5	7	2.35	0.018	0.001
W-4B	29.8	10.2	30	-5	28	1.25	0.010	0.002
S-5A	29.8	10.2	32.5	22	7	0.079	0.008	<0.001
S-5B**	29.8	10.2	32.5	22	28	0.05	0.006	<0.001
W-5A	29.8	10.2	32.5	-5	7	2.36	0.013	0.001
W-5B	29.8	10.2	32.5	-5	28	1.130	0.010	0.002
S-6A	29.8	10.2	35	22	7	0.080	0.016	<0.001
S-6B	29.8	10.2	35	22	28	0.050	0.017	0.001
W-6A	29.8	10.2	35	-5	7	2.33	0.012	0.001
W-6B	29.8	10.2	35	-5	28	1.28	0.023	0.002
S-7A	26.7	13.3	30	22	7	0.081	0.013	<0.001
S-7B	26.7	13.3	30	22	28	0.11	0.012	0.001
W-7A	26.7	13.3	30	-5	7	2.33	0.013	0.002
W-7B	26.7	13.3	30	-5	28	1.07	0.008	0.001
S-8A	26.7	13.3	32.5	22	7	0.075	0.022	<0.001
S-8B	26.7	13.3	32.5	22	28	0.10	0.023	0.001
W-8A	26.7	13.3	32.5	-5	7	2.20	0.013	<0.001
W-8B	26.7	13.3	32.5	-5	28	0.972	0.010	0.002
S-9A	26.7	13.3	35	22	7	0.065	0.009	<0.001
S-9B	26.7	13.3	35	22	28	0.15	0.006	0.001
W-9A	26.7	13.3	35	-5	7	2.28	0.013	0.002
W-9B	26.7	13.3	35	-5	28	1.42	0.013	0.002

* S-4B ranked #1 for leach-resistance
** S-5B ranked #2 for leach-resistance
† S-2A ranked #3 for leach-resistance
†† S-1B ranked #4 for leach-resistance

] based on low Se, B, As in preliminary screening extraction test.

experimentation with Consol waste and water had shown saturation pH values of ~12.4 at 22°C. This corresponds exactly to the saturation pH of $Ca(OH)_2$. All screening test extracts were analyzed for B, Se and As - elements which, based on past experience with leaching of US bituminous fly ash, have increased solubilities at high pH. The four most leach-resistant cured waste materials, ie those with the lowest extract levels of B, As and Se, were identified as S-4B, S-5B, S-2A and S-1B. These were subjected to further, more detailed leaching tests (1984 MOE protocol) and to engineering tests for evaluation as landfill materials.

MOE Leaching Test of Fixated Consol LIF Waste

The four most leach-resistant fixated waste materials identified from the preliminary screening test described above were each subjected to the MOE Leachate Extraction Procedure (October, 1984 protocol) under monofill and co-disposal anaerobic conditions. Each extract was analyzed for the same 26 species as previously determined for the MOE leachates of the 'as received' Consol LIF waste material. Results are reported in Table IX. The fixated samples had improved leach resistance over the 'as received' material: barium and boron in the fixated waste extracts both met the criteria for backfill use. Only TDS remained above the backfill requirement (~4 000 mg/L versus 2 500 mg/L). This is attributed to presence of $Ca(OH)_2$ and $CaSO_4$, which contributed to calcium hardness. As a group, the four selected fixated Consol LIF wastes had comparable leaching characteristics.

Engineering/Physical Testing of Fixated Consol LIF Wastes

To measure the 'handleability' and suitability of fixated LIF waste for landfill, each of the four selected materials from the screening tests was subjected to a set of engineering/physical tests, described in detail in the Appendix.

Handling properties of the fresh mixes were assessed by three tests:

(a) flow test (an indicator of 'pumpability')

(b) bulk-density measurement, and

(c) water content measurement

Samples S-4B and S-5B had flow test measurements of 14 cm and 16.8 cm respectably, and are probably more 'pumpable' than samples S-2A and S-1B, which had flow test measurements of 7.9 and 8.3 cm respectively. It is noteworthy that the more

TABLE IX

HOE LEACHATE EXTRACTION: ANALYTIC RESULTS*

FOR SELECTED FIXATED CONSOL LIF/FLY ASH/WATER MIXTURES

Parameter	Cured Sample S-4B		Cured Sample S-5B		Cured Sample S-2A		Cured Sample S-1B		Backfill Use: Maximum Acceptable Extract Concentration (Monofill Test)/1,2,3/	Hazardous Waste Categorization: Maximum Extract Concentration for Exemption (Co-Disposal Test)/1,2/	Comments
	Monofill	Co-Disposal	Monofill	Co-Disposal	Monofill	Co-Disposal	Monofill	Co-Disposal			
pH	12.5	12.3	12.7	12.5	12.8	12.7	12.5	12.4	-	-	No significant change in pH
As	<0.001	0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.05	5	
Ba	<1.0	1.0	<1.0	<1.0	<1.0	<1.0	<1	<1.0	1.0	100	Much lower Ba, B than Consol LIF 'as received' waste extract
B	<0.05	0.06	0.05	0.08	0.11	0.40	<0.05	0.09	5.0	500	
Cd	0.0001	0.0003	<0.0002	<0.0002	<0.0003	<0.0003	<0.0002	<0.0002	0.005	0.5	
Cr (Total)†	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	0.1	0.05	5	Cr detection limit being improved
"VAP" Cn	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04	0.2	20	
F⁻	0.4	0.7	0.5	0.80	0.5	1.00	0.4	0.9	2.4	240	
Pb	<0.004	<0.005	<0.004	<0.004	<0.005	<0.005	<0.004	<0.004	0.05	5	
Hg	<0.0002	0.0005	0.0003	<0.0002	<0.0002	0.0005	<0.0002	0.0002	0.001	0.1	
NO_3^- (as N)	0.018	0.008	0.007	0.013	0.020	0.031	0.015	0.025	10.0	1000	
NO_2^- (as N)	0.019	0.019	0.016	0.017	0.017	0.019	0.014	0.019	1.0	100	
Total [NO_3^- + NO_2^-] (as N)	0.037	0.027	0.023	0.030	0.037	0.050	0.029	0.044	10.0	1000	
U †	<0.05	<0.1	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.02	2	
Ra-226	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	0.003	0.3	U detection limit being improved
Se	0.6	0.6	0.6	0.6	0.2	0.3	0.4	0.7	3.0 pCi/L	300 pCi/L	
Ag	0.003	0.004	0.005	0.004	<0.001	<0.001	<0.001	<0.001	0.01	1	
Cl^-	0.0005	<0.0005	<0.0005	<0.0005	<0.0005	0.0031	<0.0005	<0.0005	0.05	5	Lower Se than Consol LIF 'as received'
Cu	113	133	112	127	147	154	131	146	1250	-	
Fe	<0.002	<0.002	<0.002	<0.002	<0.010	<0.010	<0.002	<0.002	5.0	-	
Mn	<0.10	<0.10	<0.10	<0.10	<0.1	<0.1	<0.1	<0.1	1.5	-	
Organic N	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.25	-	
Phenols	0.18	<0.04	<0.04	<0.04	<0.04	0.15	<0.04	<0.04	0.75	-	
$SO_4^=$	<0.001	0.004	<0.001	0.001	<0.001	0.001	0.001	0.002	0.01	-	
TDS (105°C)	1255	1009	1238	1009	1870	1540	1690	1410	2500	-	
TOC	3820	N/A	3860	N/A	4410	N/A	4450	N/A	2500	-	
Zn	2	N/A	<2	N/A	4.0	N/A	<2	N/A	25.0	-	Observed TDS levels exceed 2,500 mg/L
	<0.02	<0.020	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	25.0	-	

* All concentrations given in mg/L except Ra-226, given in pCi/L

† Analytical interference due to high background Ca concentrations

N/A Denotes not applicable due to acetic acid addition in co-disposal test

'pumpable' pair contained added fly ash, while the other two contained no added fly ash. For comparison, K-Krete, a pumpable backfill material specially developed by Ontario Hydro Civil Research Dept, has a flow measurement of 18 cm.

The bulk density of all four selected mixes were in the range 1 540 to 1 580 kg/m^3, similar to the values for loose sand/soft clay. The water contents, which were fairly similar, were in the range 53-64% (dry basis), considered to be high compared to most soils, though similar to soft marine clays.

For landfill disposal, should the fresh mix prove fluid enough to be pumped into place, minimal problems with dusting are anticipated during placement. If, on the other hand, the fresh mix is unsuitable for pumping, and has to be mechanically conveyed into place (eg, by belt conveyor) there is danger that dust-free operation during placement may be seriously compromised.

The four laboratory-cured mixes (100% relative humidity, 20°C) were subjected to three tests:

(a) shear strength test

(b) consolidation test, and

(c) permeability test

All four mixes had 15-day shear strengths of 320 kPa, resembling very strong soils, eg glacial till, or K-Krete. The consolidation test consistently yielded curve shapes resembling clay soils with pre-consolidation pressures of 580 to 1 300 kPa. Permeabilities of the four fixated materials were in the range 3.9×10^{-7} to 2.2×10^{-6} cm/s, similar to clays or glacial till. (The fly ash-containing test pair had the higher permeabilities.)

In summary, only in the pre-cure flow test was there substantial difference in physical/engineering properties. In this regard, further practical information would probably best be obtained from actual field pumping trials.

Slaking/Pelletizing Equipment Evaluation

Since dusting may prove to be a problem during handling/disposal of LIF waste, and it is at present uncertain whether equipment (eg, dustless unloaders) available on-site at Lakeview TGS or other stations will provide safe, effective means of adding water, small-scale tests of commercial slaking/pelletizing equipment have been performed.

Slaking

A 2-kg sample of Consol waste was submitted to Wallace and Tiernan, Scarborough, Ontario, for laboratory-scale slaking tests. A reply was received, stating that "the CaO will chemically combine with the silica in the fly ash ($3CaO \cdot SiO_2$) leaving only a small fraction of the original CaO to be hydrated. The results will be a very low temperature rise and an end product that will have some modest setting properties". This conclusion was surprising, since we have observed (see above) temperatures reaching ~100°C in calorimetric (slaking) experiments, and bulk slaking temperatures reaching ~160°C. The optimum temperature for paste slaking of CaO is reported/6/ to be ~88°C. We have thus far observed only plaster-like setting properties, which do not appear to reflect any high degree of combination of CaO with silica. Clarification was therefore sought from Wallace and Tiernan, and a request made for a copy of their their laboratory evaluation report. The reply received merely indicated that an 8°C rise in 6 minutes had been observed on slaking the sample in accordance with AWWA Standard Test B202-77, used to test for reactivity of quicklime (100 g) with water (400 g) at 25°C. In their opinion, half the CaO present had been slaked and the other half combined with silica present in the fly ash component. Upon further inquiry/7/ Wallace and Tiernan discouraged use of their standard slaking equipment (even for testing purposes) for several reasons, including:

(a) potential problems of abrasion associated with presence of siliceous material (specifically fly ash) in the waste, that would require extensive (and costly) equipment modifications,

(b) unduly low reaction temperatures anticipated under full-scale paste slaking conditions (in contrast with the optimum temperature of ~88°C easily attainable for slaking of regular, industrial-grade quicklime), and

(c) possible equipment plugging by pozzolanic set of the slaked LIF waste.

It is suggested that a pug mill might be more effective in slaking the LIF waste: this kind of equipment is used in blending about 4% (dry weight basis) of added quicklime (CaO) with fly ash and scrubber sludge vacuum filter cake in the fixation of natural oxidation FGD waste solids prior to disposal/8/.

Pelletizing

Pelletizing the LIF waste could prove to be an effective way of combatting handling problems anticipated for this material. A 25 kg sample of Consol LIF waste was sent to Ferro-Tech, Wyandotte, Michigan, USA, for evaluation. A semi-pilot scale test was conducted using their patented 'Turbulator' pelletizing unit, with promising results. A sample of dry, practically dust-free granular slaked product was returned to Research Division for inspection. The product material could be easily 'poured' from one container to another, and on wetting with water gave negligible temperature rise.

Ferro-Tech plan to fabricate full-scale equipment at Windsor, Ontario, thus complying with the "Made in Canada" preferred classification. Rental/purchase costs are negotiable via their Canadian representative, Steve Placek Company Ltd., Toronto, Ontario.

SUMMARY

Consol LIF waste is considered to be the closest available solid waste material to that expected to be produced in the late fall, 1985, Lakeview test of limestone injection for acid gas control. Preliminary characterization of Consol LIF waste revealed that it contained by weight about 33.3% of CaO, <3% of $Ca(OH)_2$, 18.2% of $CaSO_4$, 8% of $CaCO_3$, and 12.5% of unburnt carbon. The material reacts exothermically with water: maximum temperatures of ~100°C have been observed by calorimetry and ~160°C under bulk conditions. The heat of reaction was estimated to be about -68.9 cal/g, attributed predominantly to slaking of the CaO. Bulk slaking was accompanied by about 15% expansion. Based on MOE leachate extraction test results, the 'as received' Consol LIF waste is non-hazardous and suitable for backfill except for marginally excessive levels of barium, boron and of TDS. Particle size reduction takes place on slaking. Twenty-four to forty-eight hours after mixing with water, either with or without added fly ash, the slaked Consol waste sets to a friable, plaster-like solid. The consistency of the freshly wetted solid waste product depends on the proportion of water added. As the amount of water added is increased up to 75% w/w of water (dry solids basis) the fresh mix ranges from dry to lumpy-moist to a wet plaster-like consistency. Above 75% w/w of added water, phase separation occurs, giving a supernatant aqueous layer. The region around 75% of added water (dry weight basis) appears to be of maximum interest for handling/disposal purposes. Based on standard MOE leaching tests, the fixated waste material is non-hazardous, and is suitable for backfill except for slightly elevated TDS - attributed to dissolved $CaSO_4$ and $Ca(OH)_2$, contributors of calcium hardness in water.

Engineering/physical tests of fixated Consol LIF waste with and without added fly ash indicate that the products resemble natural earth materials. Only in flow characteristics do the freshly mixed LIF/fly ash/water blends differ substantially, and some differences in 'pumpability' into place are anticipated. Should use of reduced proportions of added water be seriously considered, it is important to recognize that this would tend to increase dusting during the fixation process.

Highly promising results were obtained from preliminary commercial pelletizing tests of Consol LIF waste: the slaked product was granular and dust-free. A lime-slaking equipment manufacturer performed laboratory testing of Consol LIF waste, and were discouraging of use of their equipment for this kind of application. Use of pug mills for slaking limestone injection solid wastes may prove to be a more effective approach.

RECOMMENDATIONS

1. It is recommended that care be exercised in handling/disposal of LIF waste in view of its potential for exothermic reaction, expansion and dusting.

2. Further work is recommended in the following areas:

   (i) Laboratory characterization of Lakeview LIF waste, similar to that done for Consol LIF waste.

   (ii) Laboratory evaluation of other LIF wastes produced under varying operating conditions, eg Ca/S ratio, alternative sorbents and promoters, etc, as necessary.

   (iii) Scale-up work on pelletizing, waste fixation trials, dry handling/disposal methods, etc., as necessary.

   (iv) Tests of using a pug mill for slaking LIF waste with water.

(v) Tests of disposal by pumping the fresh LIF waste/fly ash/water mixes into place in a dyked-off area where it is allowed to set and cure.

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APPENDIX

PHYSICAL/ENGINEERING PROPERTIES OF CONSOL LIF FIXATED WASTE

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PHYSICAL PROPERTY TESTING OF LIMESTONE INJECTION FEED (LIF) MATERIALS

C.B.H. Cragg

INTRODUCTION

As an alternative to wet scrubbing the flue gases of coal fired generating plants, finely ground limestone can be injected into the boiler, with the coal, to remove SO_2 and NO_x by a dry process. The by-product of this limestone injection feed (LIF) process, consisting of roughly equal parts of fly ash, gypsum and free lime, is collected on the precipitators prior to disposal. Earlier testing by Chemical Research found that LIF, alone or in combination with fly ash, gave the best leachate and dust performance when mixed with sufficient water to form a wet paste consistency and allowed to cure. Four mixes of fly ash, LIF solid waste and water having the best performance in these tests were selected by Chemical Research for physical property testing. These tests and their results are described.

TESTS PERFORMED

At the time of mixing the samples, slump tests and flow tests were done. These tests are normally used for materials such as concrete or K-Krete (a cement stabilized soil) to determine pumpability and product quality.

After the samples had been allowed to harden and cure, strength, compressibility and permeability properties were measured. Unconfined compression tests, consolidation tests and triaxial permeability tests, normally used for soil evaluation, were employed for these property measurements. Sample bulk density and water content (dry basis) determinations were also made in the course of these tests.

SAMPLE PREPARATION

Sample ingredients were LIF waste (supplied by Chemical Research), fly ash (from Lakeview TGS) and Toronto tap water. The mix proportions were as follows:

job	file	date	report no.
740631-232-00490	843.2	November 20, 1985	S85-33-K

Mix No	LIF (g)	Fly Ash (g)	Water (g)	Reference Mix
1	29.8	10.2	30.0	S-4B
2	29.8	10.2	32.5	S-5B
3	40.0	0	32.5	S-2A
4	40.0	0	30.0	S-1B

Ingredients were blended in a Hobart mixer to a uniform consistency, then poured and rodded into sample molds. The samples were cured in a moist room at 20°C and 100% relative humidity until tested. In general, the length of curing before physical property testing was 15 days.

TEST RESULTS

Material Handling Properties

The testing was performed in July and August of this year. Table I shows the testing schedule and the results of flow tests done at the time of sample preparation. Flows ranged from 7.9 cm to 16.8 cm. Mixes containing fly ash had flows about twice that of mixes without fly ash. For comparison, a flow of about 18 cm is normally required to obtain a pumpable K-Krete mix that does not segregate. Lower flow material, particularly material containing fly ash, can be pumped. It is anticipated that Mixes 1 and 2 will be pumpable but Mixes 3 and 4 may not pump without increased water contents. However, full scale pumping tests should be performed to determine the pumpability of all these mixes.

All mixes had similar values for bulk density and water content. Average (per mix) bulk density ranged from 1540 to 1580 kg/m^3. Dry basis water contents had average values from 53% to 64%. The density values are similar to those for loose sand or soft clay. The water contents are not typical of soils, though such high values are found in soft marine clays and even higher values are measured in peat.

Strength Properties

Detailed results of the shear strength, consolidation and permeability tests are grouped in Table II. Shear strength tests were performed on samples aged 8 to 45 days which caused some scatter in the results. To permit a comparison, shear strength versus age at testing is plotted in Figure 1. It may be seen from this graph that the LIF test mixes had quite similar shear strengths at an age of 15 days. These strength values are similar to very strong soils, such as dense glacial till, or to soil cements, such as K-Krete.

The consolidation tests show (Table II) that the test specimens had compressed from 8% to 21% under a pressure of 3000 kPa. Individual graphs for the consolidation tests are shown in Appendix I. These curves have a "knee" characteristic of preconsolidated clays, with preconsolidation

pressures ranging from 450 kPa to 1300 kPa. The preconsolidation pressure is the pressure that can normally be safely applied to a soil without causing large settlement.

Permeability values for the LIF mixes ranged from 3.9×10^{-7} cm/sec to 2.2×10^{-6} cm/sec. The two highest values occurred in the mixes with fly ash. These results have been averaged with the other results describing these mixes even though they may be due to sample disturbance or test process instead of material differences. Apart from these values, all results were remarkably uniform. These very low permeabilities are typical of clay or clayey glacial till soils.

Comparison of Mix Properties

Properties of the four LIF mixes studied are summarized in "average" form in Table III. No significant differences were observed in the behaviour of these mixes except that the fly ash mixes (Mix 1 and 2) had better flow properties. Higher permeabilities recorded for two of four tests on fly ash mixes may or may not be properties of the mix. The permeability difference is a minor one.

SUMMARY

Physical property and strength tests were performed on four mixtures of LIF, fly ash and water selected by Chemical Research. The mixes incorporating fly ash had better flow properties suggesting they are more conducive to being pumped into place. Field pumping trials are recommended to confirm pumping properties of the mixes. Apart from the flow properties, the mixes were essentially equal in all other properties measured. The values obtained have been described in terms of soils with similar property values, for clarity.

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TABLE I

Test Sequence and Flow Properties

Mix No	Ref Mix	Date Poured 1985	Minutes After Mixing	Flow cm	Slump cm	PERMEABILITY No	PERMEABILITY Date	CONSOLIDATION No	CONSOLIDATION Date	U/C No	U/C Date
1	S-4B	June 10	2 5	13.5 14.5	9.0 7.5	2	June 25	2	June 25		-
2	S-5B	June 17	3	16.8	10.5	2	July 2	2	July 2	1	July 2
3	S-2A	July 19	3	7.9	2.1	2	July 26	2	July 26	2	Aug 1
4	S-1B	July 31	3	8.3	2.3	2	Aug 15	2	Aug 15	2	Aug 15

TABLE II

Strength Test Results

Mix No	Sample Age (days)	Shear Strength (kPa)	Failure Strain (%)	Consolidation Test Net Strain (%)	Permeability Test Permeability (cm/sec)
1	15			8.3	
	15			13.1	
	15				5.1×10^{-7}
	15				1.6×10^{-6}*
2	8	221	2.2		
	45	730	0.8		
	15			16.9	
	15			20.9	
	15				3.3×10^{-7}
	15				2.2×10^{-6}
3	13	271	3.0		
	13	236	3.0		
	7			14.2	
	7			13.5	
	7				9.6×10^{-7}
	7				3.9×10^{-7}
4	15	301	1.9		
	15	350	1.0		
	15	395	1.2		
	15			9.4	
	15			11.0	
	15				3.9×10^{-7}
	15				4.2×10^{-7}

*sample cracked after test

TABLE III

Average Properties of LIF Mixes

Mix No	Bulk Density (kg/m^3)	Water Content (%)	Flow at 3 m in (cm)	Shear Strength at 15 days age (kPa)	Consolidation Net Strain (%)	Permeability (cm/sec)
1	1540	55	14.0	-	10	1×10^{-6}
2	1560	64	16.8	300	19	1×10^{-6}
3	1580	59	7.9	250	14	7×10^{-7}
4	1550	53	8.3	350	10	4×10^{-7}

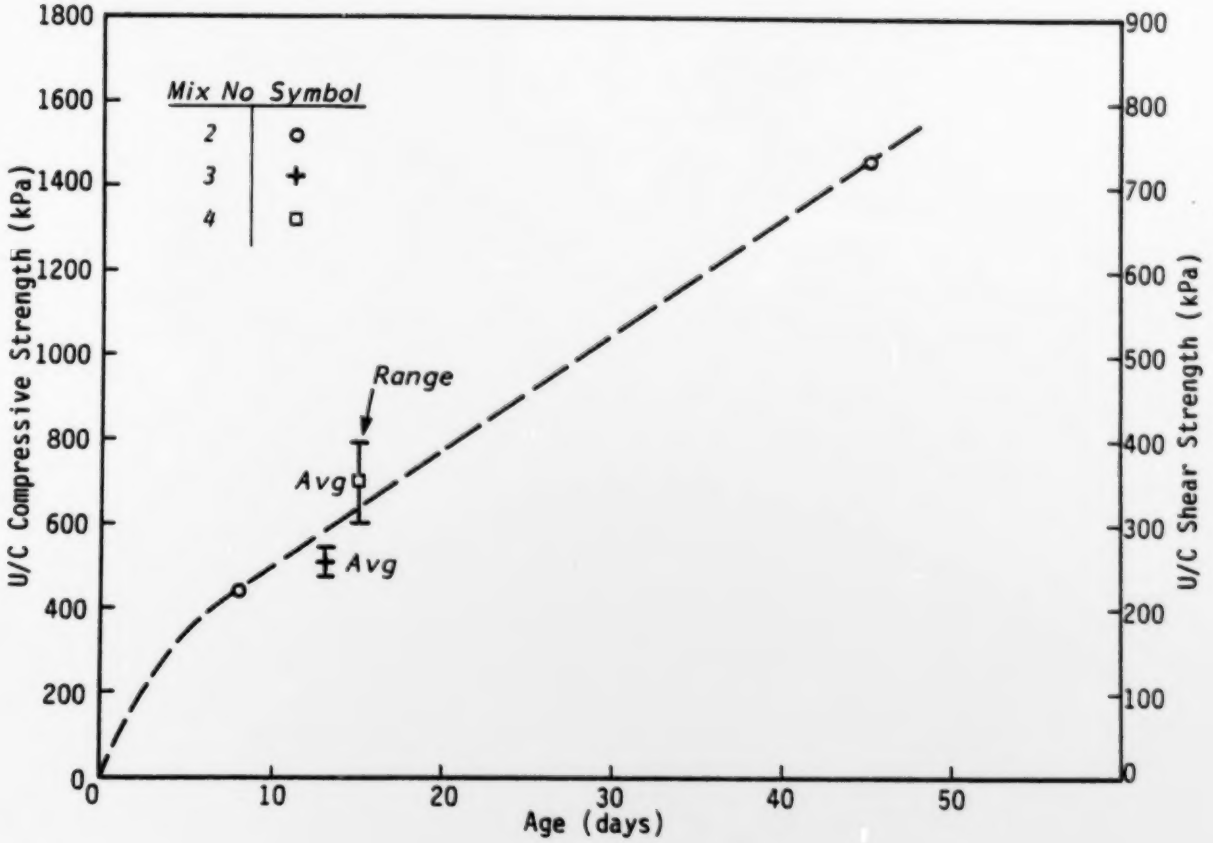

FIGURE 1
SHEAR STRENGTH VS AGE OF LIF MIXES

APPENDIX A

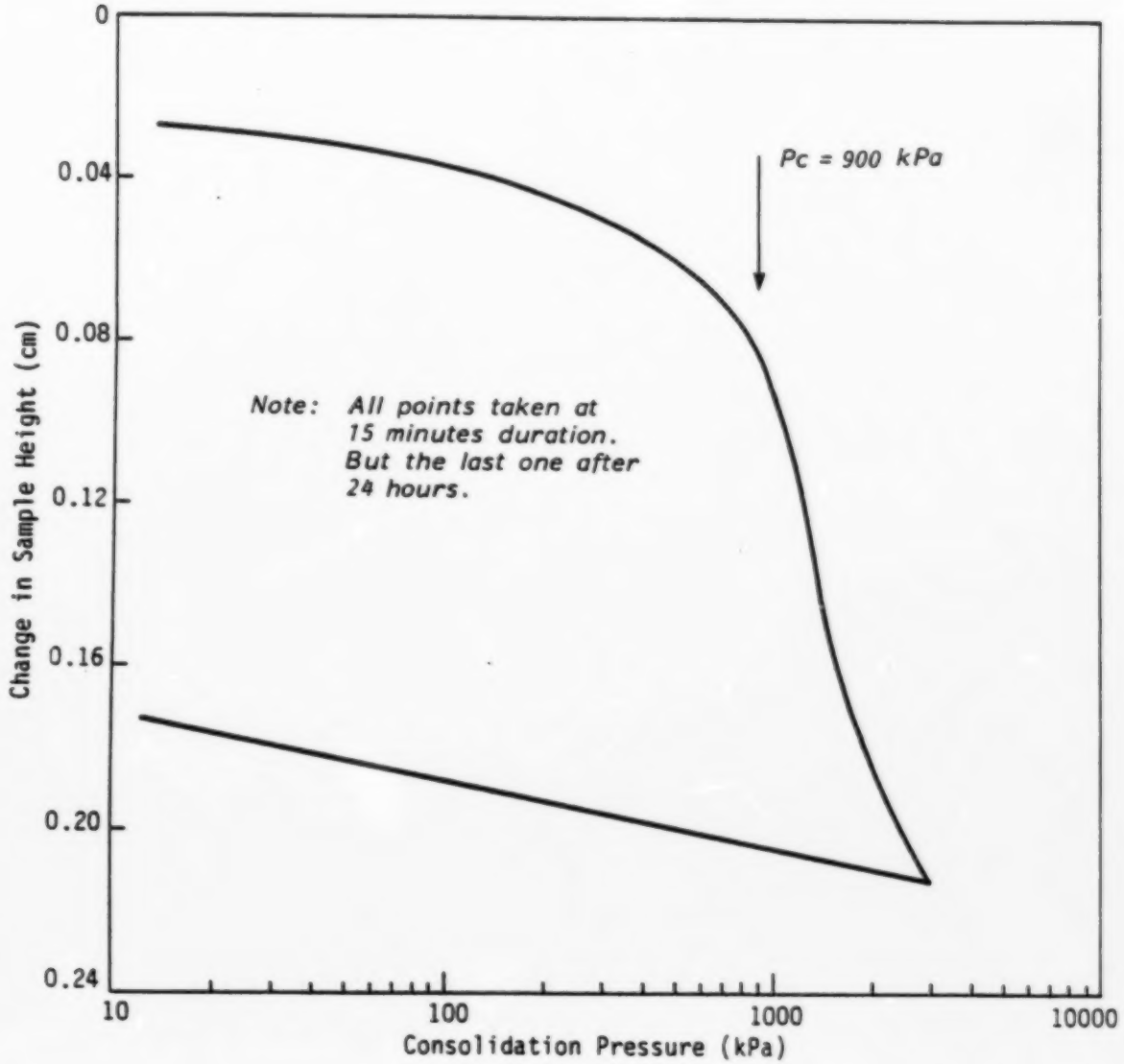

FIGURE A1

MIX #1 TEST #1
CONSOLIDATION TEST ON LIF

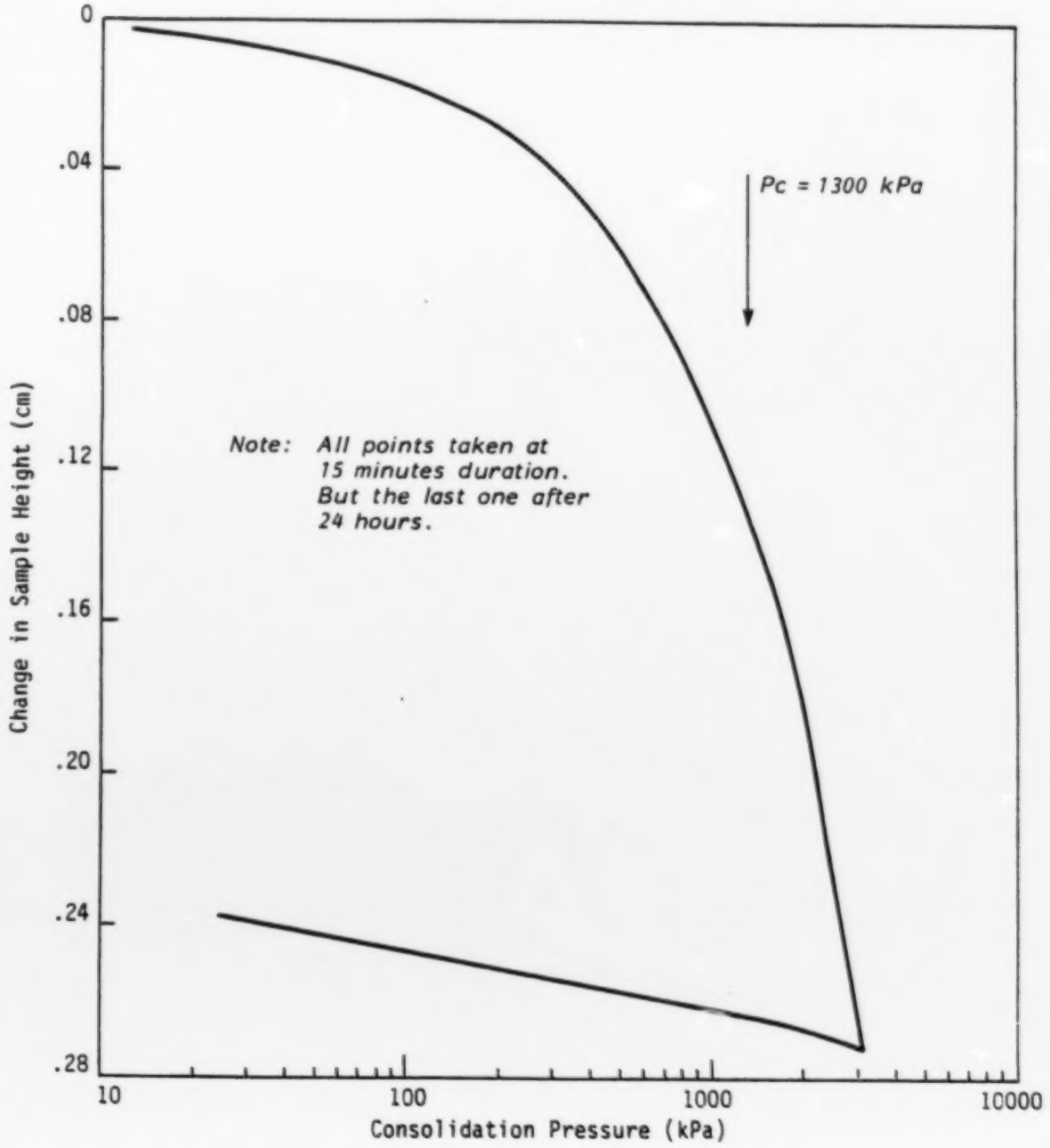

FIGURE A2

MIX #1 TEST #2
CONSOLIDATION TEST ON LIF

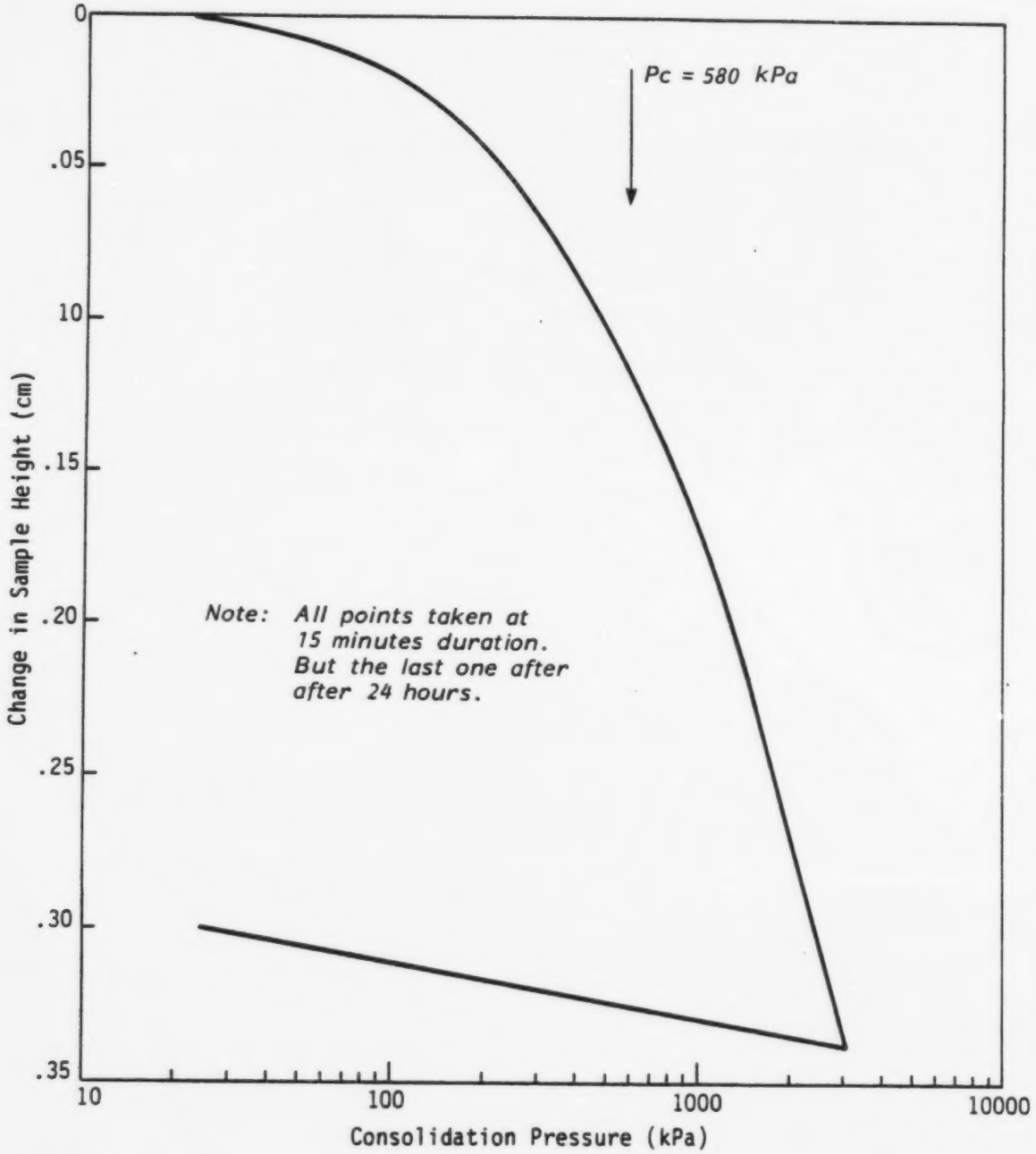

FIGURE A3

MIX #2 TEST #1
CONSOLIDATION TEST ON LIF

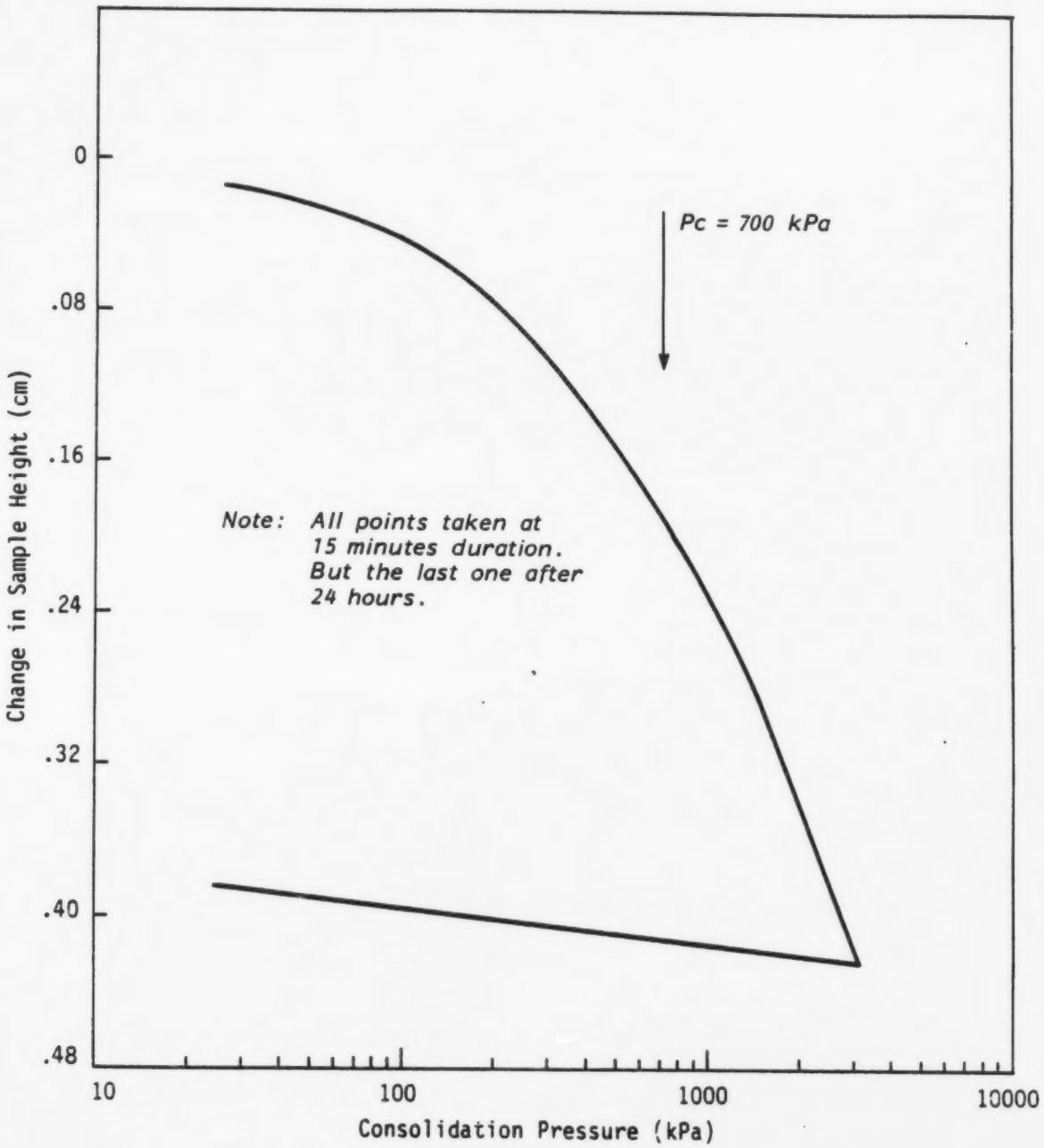

FIGURE A4

MIX #2 TEST #2
CONSOLIDATION TEST ON LIF

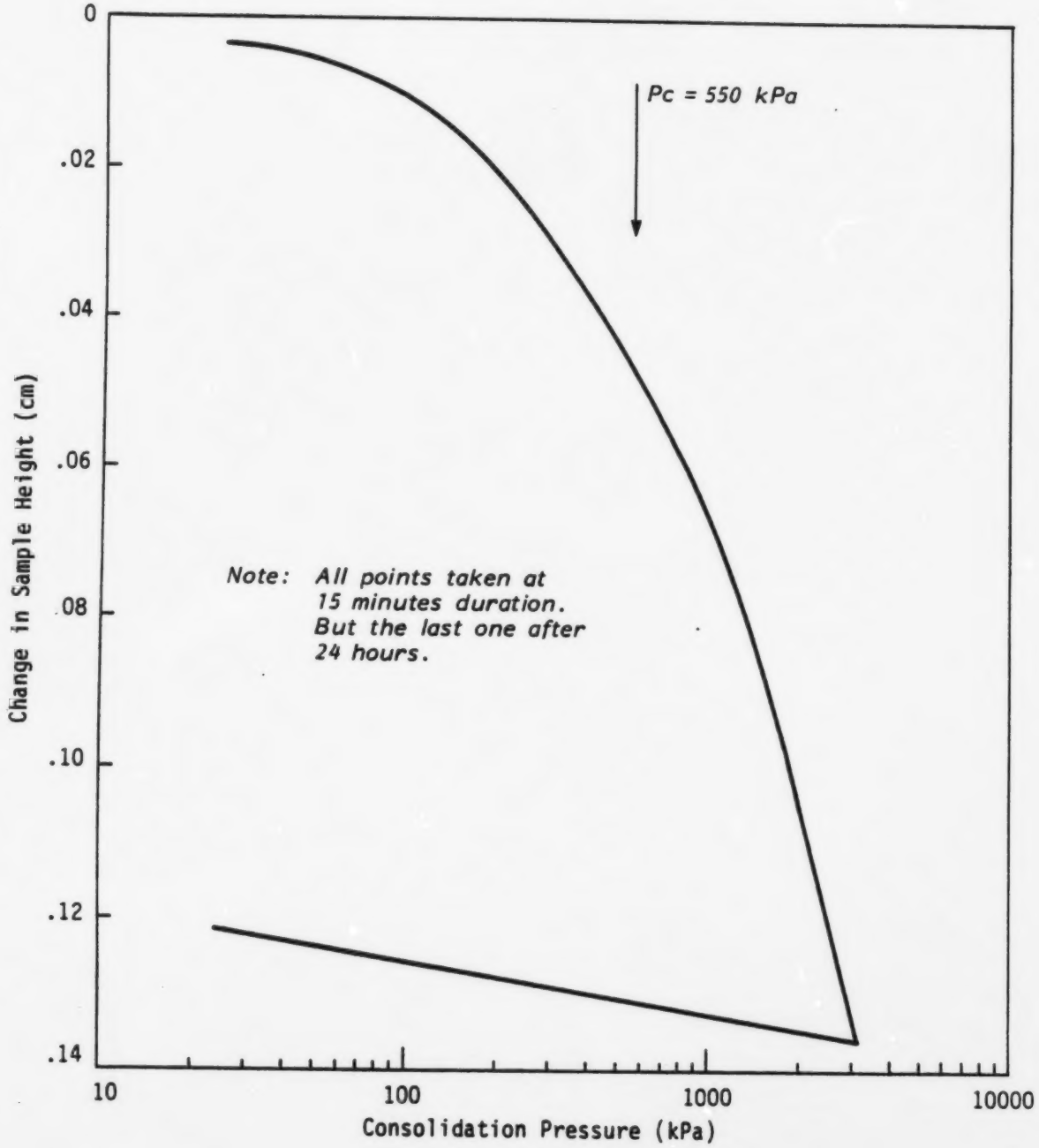

FIGURE A5

MIX #2 TEST #3
CONSOLIDATION TEST ON LIF

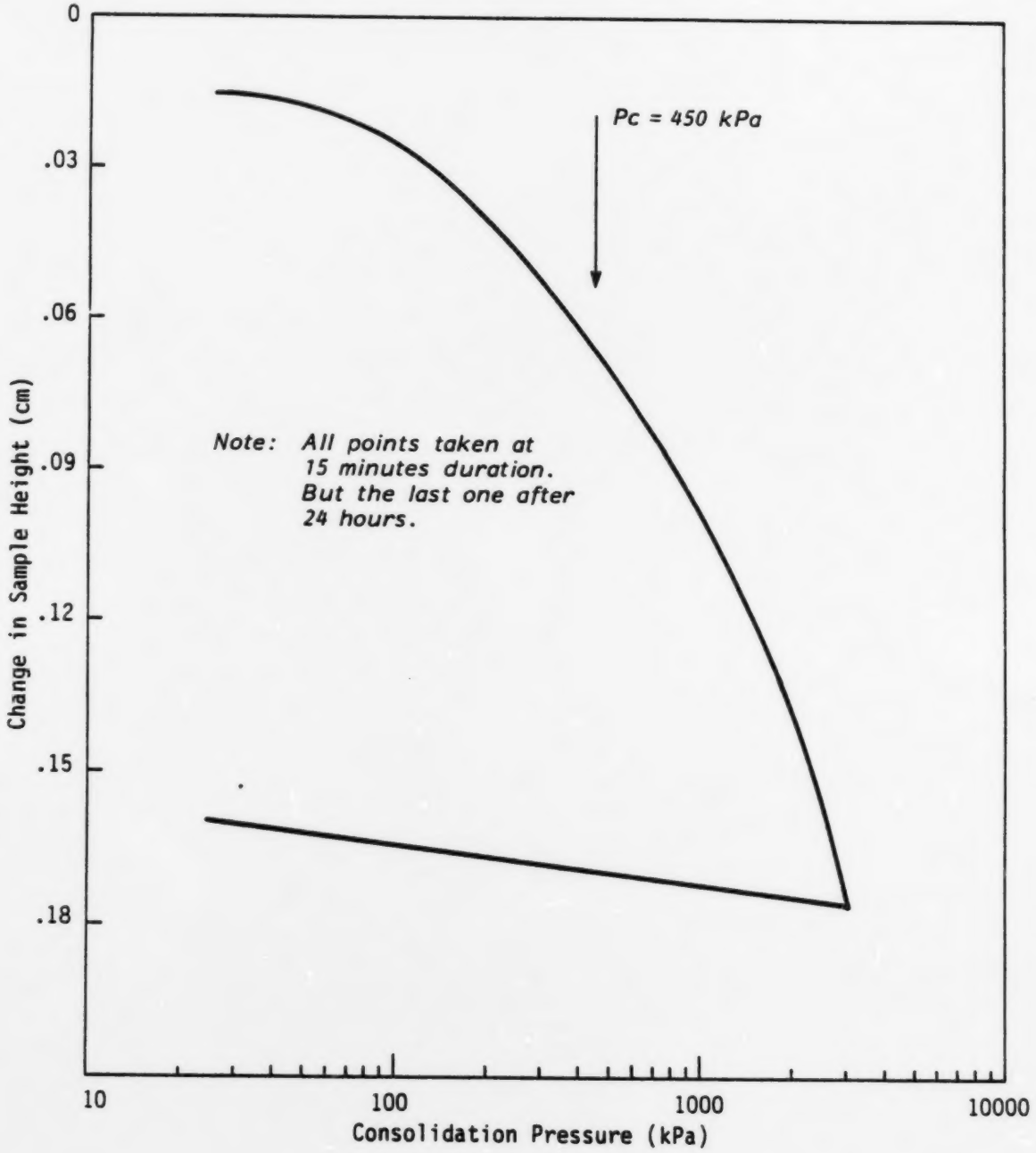

FIGURE A6

MIX #2 TEST #4
CONSOLIDATION TEST ON LIF

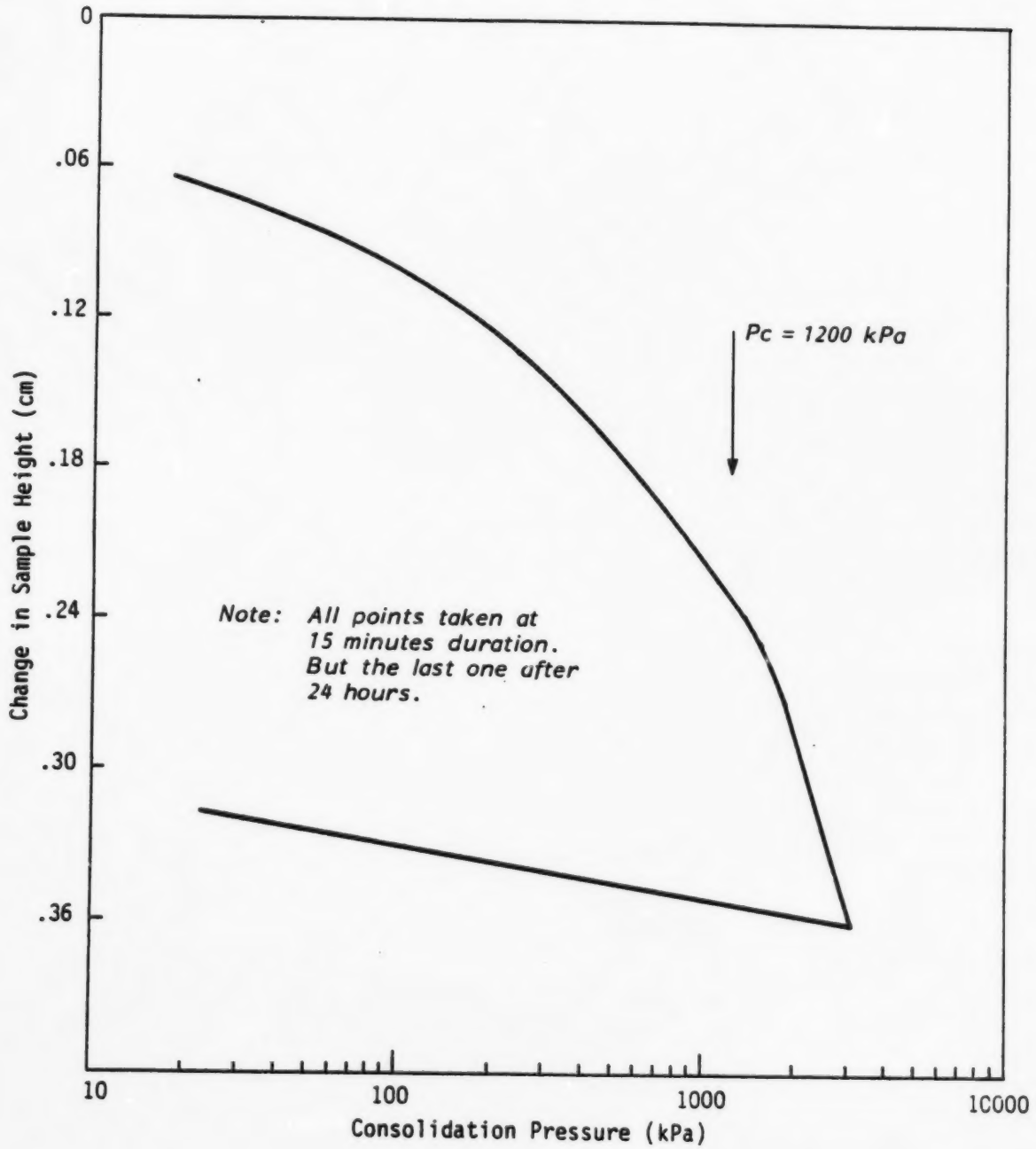

FIGURE A7

MIX #3 TEST #1
CONSOLIDATION TEST ON LIF

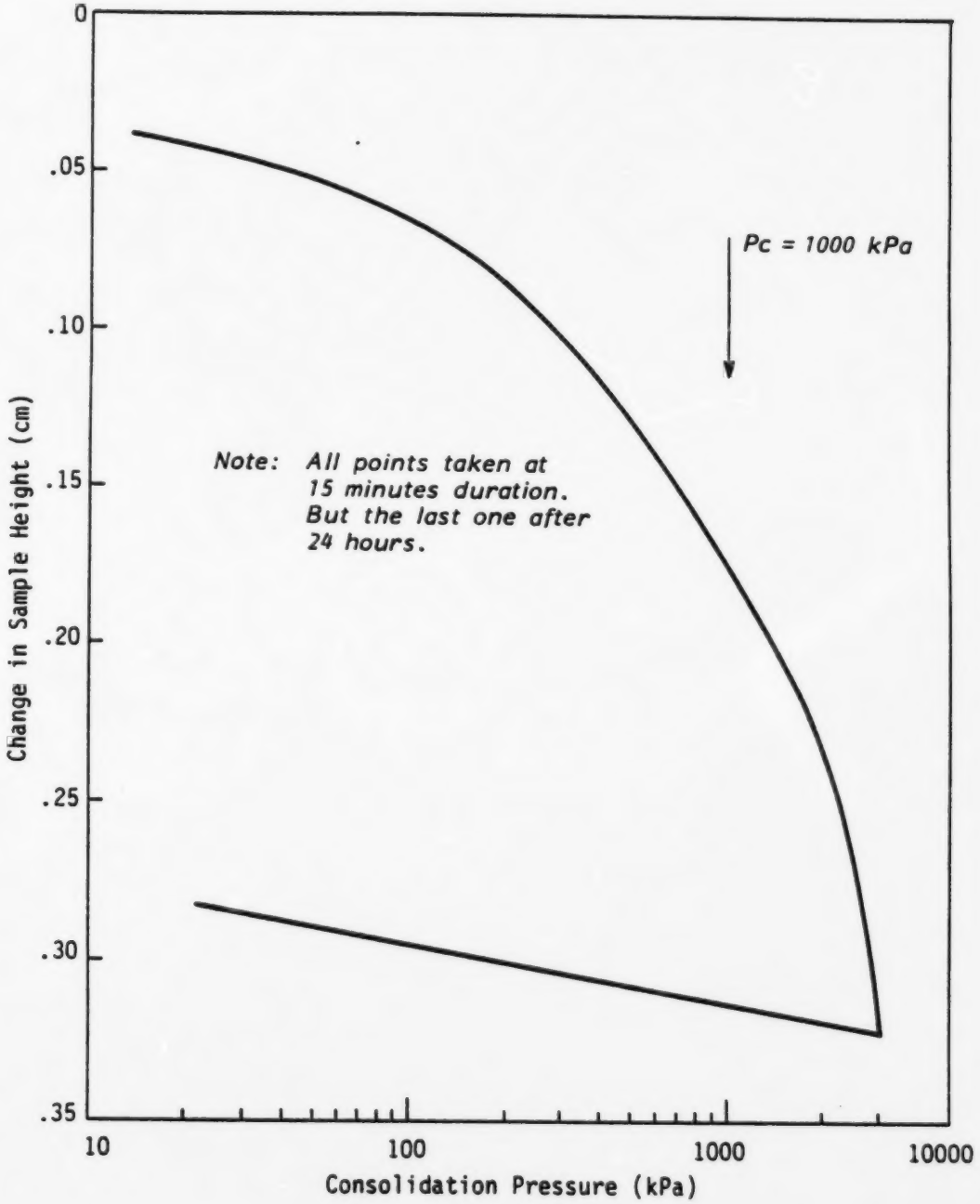

FIGURE A8

MIX #3 TEST #2
CONSOLIDATION TEST ON LIF

85-340
170016 RD

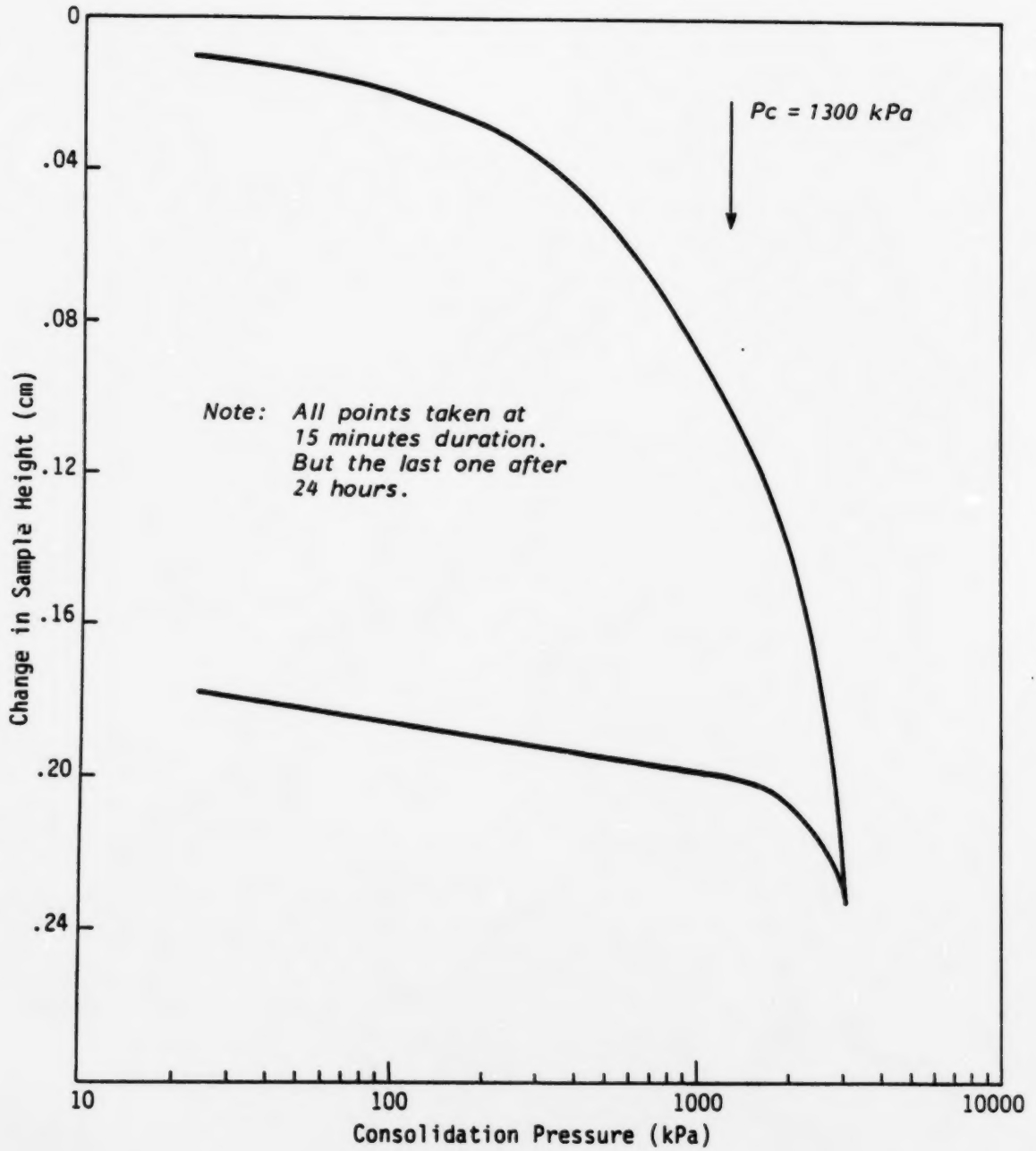

FIGURE A9

MIX #4 TEST #1
CONSOLIDATION TEST ON LIF

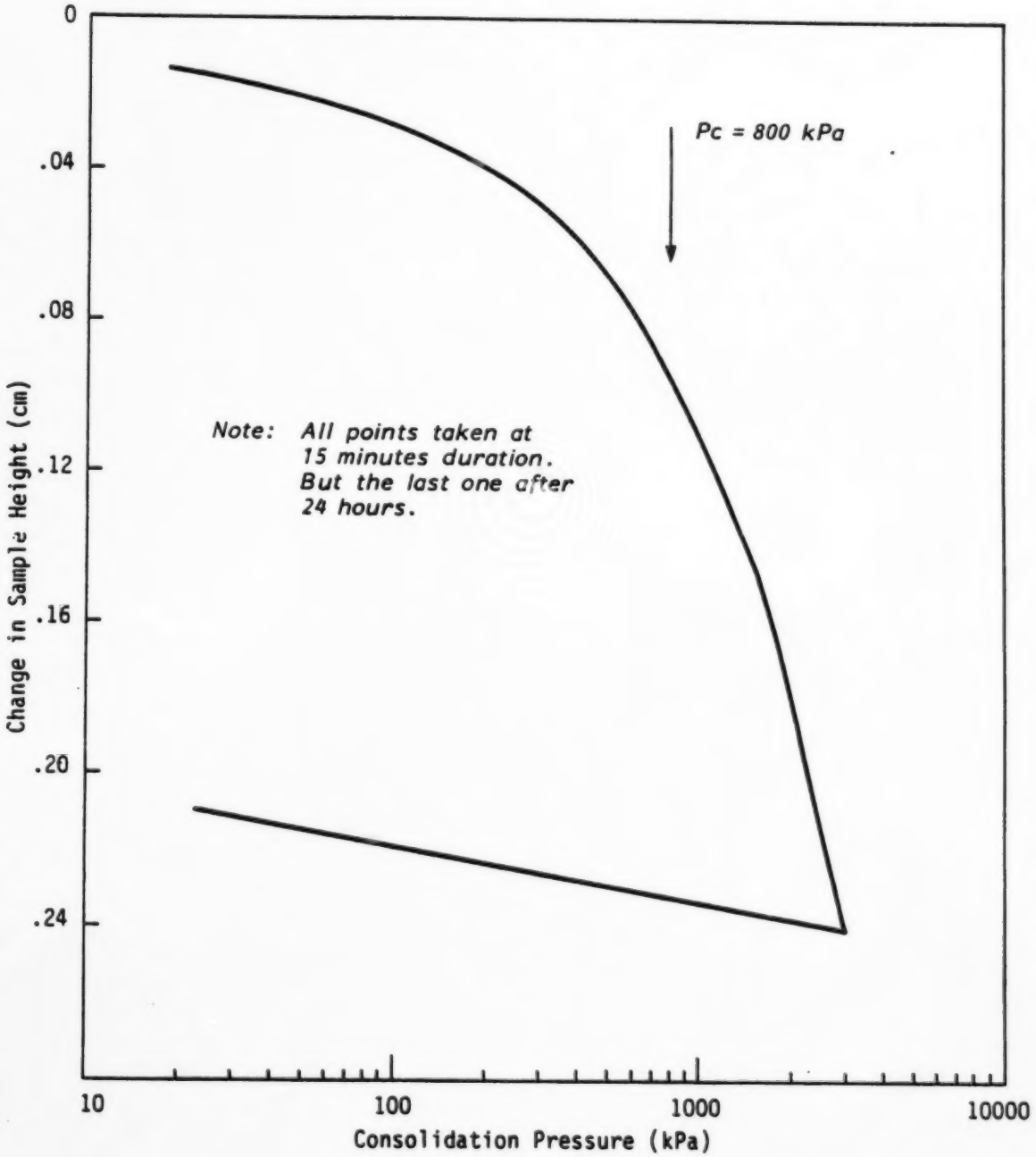

FIGURE A10

MIX #4 TEST #2
CONSOLIDATION TEST ON LIF